PATENT SPECIFICATION

1,000,044

1,000,044

Date of Application and filing Complete Specification: May 24, 1962. *No. 20051/62*

Application made in Germany (No. H42979ixb/42l) on June 28, 1961.

Complete Specification Published: August 4, 1965.

Index at Acceptance:—**G1** A (20Y, 21X, 21Y, 24Y, 26Y, 35X, 35Y, 36Y, 42Y, 43Y, 44X, 44Y, 45Y, 203, 211, 218, 242, 265, 269, 316, 365, 369, 400, 404, 406, 407, 422, 423, 426, 428, 432, 433, 436, 438, 449, 452, 459, 469, 471, 482, 487, 577, 578, 579, 621).

Int. Cl.:—G 08 c.

COMPLETE SPECIFICATION

DRAWINGS ATTACHED

Improvements in or relating to the Testing of Metals

We, HOESCH AKTIENGESELLSCHAFT of 12, Eberhardstrasse, Dortmund, Germany, a German Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

The present invention relates to a method for determining chemical elements in metal articles or in melts, based on the principle of spectrum analysis of γ-radiation released by neutron irradiation.

In order to determine the elements present in metals, use has hitherto been made of chemical analysis methods, photo-optical analysis, Röntgen fluorescence analysis, mass spectrum analysis and activation analysis.

Observation by the latter analysis methods can only be effected in practice on quite small regions of the test material. This is quite unsatisfactory insofar as basically no homogeneous material exists. With activation analysis hitherto, the test sample was irradiated in a reactor with slow, so-called thermal neutrons, making radioactive the separate elements contained therein. The activated elements then emit an electromagnetic wave radiation and/or corpuscular radiation according to characteristic decay laws, and the radiations are then measured and evaluated.

This last method, despite its great advantages over previous methods, still has many disadvantages. Hitherto the test pieces generally had to be irradiated in a reactor, since the usual laboratory safe neutron sources which can be derived from e.g. a Po-Be reaction, have in general too small a neutron flux density. On the other hand, certain elements which are very important in steel analysis, especially oxygen, are only slightly activated by slow neutrons. Finally, the irradiated test piece, namely the metal body, still remains radioactive after testing with slow neutron irradiation.

However metal bodies have also been irradiated with slow neutrons using as source a deuterium-tritium reaction of an accelerator. By this d-t-reaction, fast neutrons in the MeV energy range are produced and these are transformed into slow neutrons, e.g. by a decelerating layer of paraffin. Basically the same disadvantages appear with this method as with reactor radiation.

Furthermore it is also well known, from mineral oil mining, how to determine the presence of certain elements in the interior of the earth by means of prompt γ-radiation which is released by fast neutrons and subsequently measured and evaluated.

According to the present invention, a method is provided for the determination of chemical elements in metal bodies or melts, in which at least part of the metal body or melt is bombarded with fast neutrons and the prompt γ-radiation released by the neutrons is measured and evaluated.

This new method has several advantages over the known methods of analysis based on the principle of spectrum analysis of radioactive radiation released by slow or decelerated neutrons. Above all, it makes possible the detection of oxygen, which is very important in metallurgy. In addition, this method produces hardly any activation of the test material and consequently hardly any radioactivity. Furthermore, because of the greater depth of penetration of the fast neutrons in combination with the penetrability of the prompt γ-radiation produced thereby, it

is also possible according to this method to analyse test pieces of greater thickness. Finally this method facilitates the determination of elements in the melt itself, which has hitherto been impossible.

In order to be able to determine, in particular, the existence of oxygen, the use of a d-t-reaction is recommended to produce fast neutrons, since of all the known fast neutron producing reactions only the d-t-reaction produces neutrons with sufficient energy for releasing prompt γ-radiation from oxygen. Naturally, for other elements, a different nuclear reaction can be used by which such fast neutrons are produced that the characteristic γ-radiation of the element under analysis is stimulated.

In order to identify and/or estimate the quantity of a single element, it is necessary to determine the energy distribution of the γ-radiation produced, so that measurement is thus effected according to energy levels. Measurement of the γ-radiation may also be made according to emission groups, however; for example, it may be desirable to determine the total non-metallic contents of an alloy and, as each of the respective elements emits γ-radiation of characteristic energy levels, these characteristic levels can be measured together in what is known as an "emission group." The measurement is carried out, for example, by a pulse height analyser coupled to the outlet of a radiation detector.

On analysing certain regions of the test material, it may be necessary to filter out the γ-radiation from other regions, in particular, the γ-radiation coming from the measuring system or from the media between the sample and the detector or from other regions of the text material. Preferably this can be carried out with collimators and/or masking systems mounted on one or more radiation detectors. However, it is also possible to arrange a neutron source and two or more radiation detectors in a tandem or telescopic arrangement connected with a coincidence circuit, so that only the γ-radiation which is incident simultaneously on the different radiation detectors is measured. The spatial cross-section of the assumed cone of neutron emission and γ-ray reception therefore determines the region of the test material being analysed.

In analysing large surfaces, it is preferable to start with a neutron source which has a cone of emission irradiating the entire test surface. The radiation detector may have a cone of reception including the entire test surfaces, but it may also have a smaller cone of reception and preferably may be guided continuously e.g. in raster form, over the test surface. Also the reverse arrangement of a scanning neutron source with a narrow cone of emission and a radiation detector with a fixed cone of reception including the entire test surface is possible. Furthermore, it is possible to allow a neutron source-radiation detector system with a small measuring range to scan a large test surface, preferably continuously. Finally, it is also possible to arrange plurality of neutron sources and radiation detectors adjacent one another. If necessary, it is possible to vary the size of the cone of emission as well as the cone of reception, so that the cones can be adjusted to the actual size of the test surfaces.

In analysing melts, e.g. in metallurgical furnaces or in casting ladles, it can be interesting to ascertain the proportion of certain elements in the metal bath as well as in the slag. This can be effected by adjustment of the elevation of the measuring system or by its pivotation about a horizontal axis, so that the point of measurement shifts from the metal bath to the slag and vice versa. It is also possible to make either the neutron source or the radiation detector movable and to make immovable the other part of the measuring system having a correspondingly large cone of emission or cone of reception. One possible form of indirect comparative measurement is if the neutron source, acting on the adjoining parts of the metal bath and slag, co-operates with one radiation detector directed on the metal bath and one directed on the slag zone.

The large depth of penetration of the fast neutrons as well as the penetrability of the prompt γ-radiation released thereby readily allows the measuring system to be set up outside a metallurgical furnace, such as a Siemens-Martin furnace.

The method according to the invention is suitable particularly for analysis during operation of the furnace and working of the metal, since results of the analysis can be continually and immediately read off after a first calibration.

Its use in practice is conceivable in all finishing steps in steel mills, beginning with the analysis of the melt, through the analysis of the intermediate product and up to the analysis of the finished product, and moreover during the finishing process itself. Basically the method can be carried out continuously or repeated at timed intervals.

The following is an example of an analysis for oxygen content:

A steel test piece of 100 grams in weight was bombarded with fast neutrons originating from a d-t-source. The pulse height analyser adjusted with its channels to measure γ-rays from 4.5 to 6.7 MeV energy measures the characteristics prompt γ-radiation occurring on interaction between these neutrons and the oxygen. The radiation detector counted 500 effective impulses in a period of for example 10 seconds, i.e. impulses free from foreign radiation. From a calibration curve determined by compara-

tive analyses or comparative measurements, it was seen that there was an oxygen content of 50 mg., corresponding to 0.05%.

It is clearly possible to determine an oxygen content even in the region of 0.001% with approximately 10% relative accuracy.

The method according to the invention can moreover be used advantageously for the non-destructive testing of metal with non-metallic inclusions or for locating shrinkage holes. As opposed to known methods for the non-destructive testing of metal bodies, as e.g. the ultrasonic testing method and the testing methods based on the radiation by Röntgen γ-radiation, the present method brings to light evidence of nonhomogeneity of the body and above all of its chemical state, on the basis of determining certain elements and if necessary their concentration.

According to the present method, it is readily possible for example to determine whether a shrink hole is clean or whether the inclusions connected with the welding contain e.g. oxide, sulphide or silicate.

The following is an example:

A hot unworked steel slab bloom rolled in an ingot slab bloom frame with the dimensions 105C mm. width, 150 mm. thickness and approx. 6000 mm. length, is bombarded with fast neutrons in a measuring unit before its head and tail are cut clean. The device continuously determines the amount of oxygen, sulphur and silicon therein. To do this for example, a measuring region of 100 mm. thickness, 20 mm. width and 20 mm. length is masked and this measuring region scans approx. 70% of the slab bloom relative to its width.

A multi-channel pulse height analyser gives the count for oxygen, sulphur and silicon in the tail of the slab bloom which can be compared with corresponding calibration curves showing the normal percentages of these elements in steel. These count values vary on scanning the slab bloom only immaterially until the measuring region scans a zone at approx. 1000 mm. distant from the head end of the slab bloom, the oxygen radiation intensity of which zone rises irregularly to approx. three times the amount of the normal value. This zone widens towards the head end of the slab bloom, and the intensity of oxygen radiation from the zone varies between three and ten times the value of the normal intensity. The same holds for the increase in the sulphur and silicon values.

The excess of oxygen, sulphur and/or silicon is evidence of the existence of a shrink hole at the head filled with oxides, sulphides and/or silicates, which hole because of the slag inclusions will not weld together with further rolling and preferably should be cut off as soon as possible.

In order that the invention may be more readily understood, the invention is described below in conjunction with the accompanying drawings, in which:

Fig. 1 shows schematically a test piece being irradiated by a radiation source and a radiation detector directed on the test piece;

Fig. 2 shows an arrangement similar to Fig. 1 with a pulse height analyser coupled to the output of the radiation detector;

Fig. 3 shows an arrangement similar to Fig. 1 with the radiation detector provided with a collimator attachment;

Fig. 4 shows an arrangement similar to Fig. 1 with several radiation detectors in a coincidence circuit;

Fig. 5 shows an arrangement similar to Fig. 1 with a radiation source and a radiation detector whose cone of emission and cone of reception respectively correspond to the size of the test piece;

Fig. 6 shows an arrangement similar to Fig. 1 with a radiation source whose cone of emission corresponds to the size of the test piece and a radiation detector movable over the test piece and having an area of reception for parallel radiation;

Fig. 7 shows an arrangement similar to Fig. 1 with a movable radiation source and a movable radiation detector;

Fig. 8 shows an arrangement similar to Fig. 1 with a plurality of radiation sources and radiation detectors;

Fig. 9 shows a cross-section through a metallurgical furnace with the melt which is being irradiated by a movable radiation source and on to which a movable radiation detector is directed;

Fig. 10 shows an arrangement similar to Fig. 9 with a radiation source simultaneously irradiating the metal bath and the slag and a movable radiation detector;

Fig. 11 shows an arrangement similar to Fig. 9 with a radiation source simultaneously irradiating the metal bath and the slag and having a radiation detector directed on the metal bath and a radiation detector directed on the slag.

Figs. 1 - 8 show a test piece 11 and its analysis zone 12, the source of radiation 13, a radiation source 113 with a large cone of emission, a movable radiation source 213, the radiation detector 14, a radiation detector 114 with a large cone of reception, a movable radiation detector 214, a pulse height analyser 16, a collimator 17 and an indicating or registering apparatus 18.

Figs. 9-11 show also a metallurgical furnace 9, with the slag 111 and the metal bath 211.

The separate known components of the device are shown quite schematically, partly by their standard symbols. Only the effective cross-section of the cones of emission and reception of the radiation source and radiation detector respectively are shown.

WHAT WE CLAIM IS:—

1. A method for the determination of chemical elements in metal bodies or melts, in which at least part of the metal body or melt is bombarded with fast neutrons and the prompt γ-radiation released by the neutrons is measured and evaluated.

2. A method as claimed in claim 1, in which the fast neutrons are produced by d-t-reaction.

3. A method as claimed in claim 1 or 2, in which the prompt γ-radiation is measured according to energy levels.

4. A method as claimed in claim 1 or 2, in which the prompt γ-radiation is measured according to emission groups.

5. A method as claimed in any preceding claim, in which the regions of the test material other than those to be analysed and the prompt γ-radiation, originating from other media are masked out.

6. A method for the determination of chemical elements in metal bodies or melts based on the principle of spectrum analysis of γ-radiation released by neutron irradiation, as hereinbefore described with reference to Fig. 1, Fig. 2, Fig. 3, Fig. 4, Fig. 5, Fig. 6, Fig. 7, Fig. 8, Fig. 9, Fig. 10 or Fig. 11 of the accompanying drawings.

POLLAK, MERCER & TENCH,
Chartered Patent Agents,
Audrey House, Ely Place,
London, E.C.1.
Agents for the Applicants.

Berwick-upon-Tweed: Printed for Her Majesty's Stationery Office by The Tweeddale Press Ltd.—1965
Published at The Patent Office, 25 Southampton Buildings. London. W.C.2 from which copies may be obtained.

1,000,044 COMPLETE SPECIFICATION
3 SHEETS

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SHEET 1

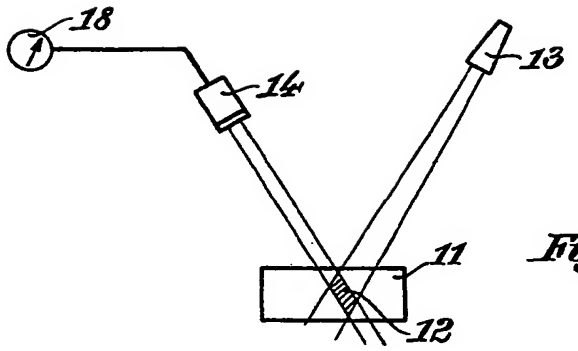

Fig. 1

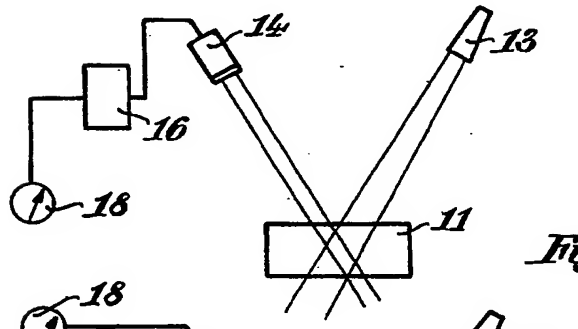

Fig. 2

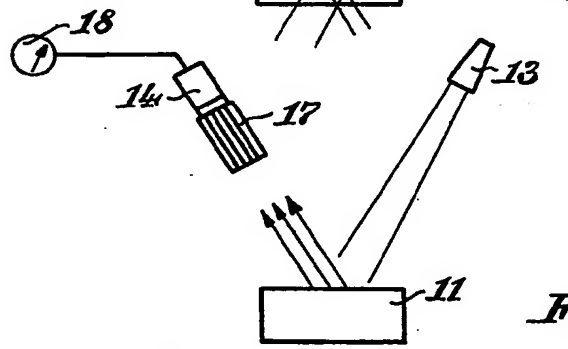

Fig. 3

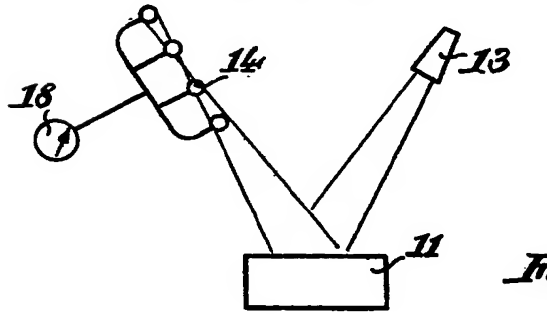

Fig. 4

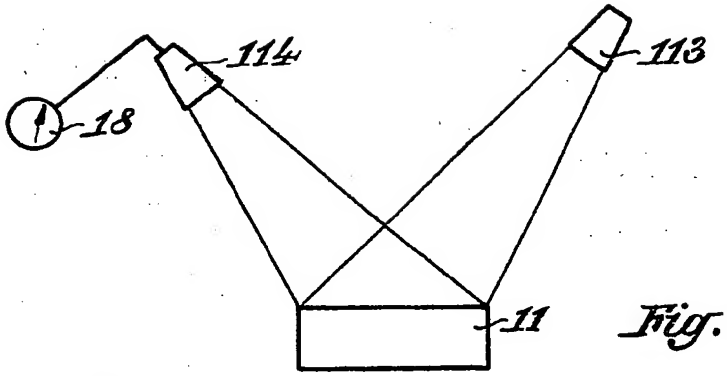

Fig. 5

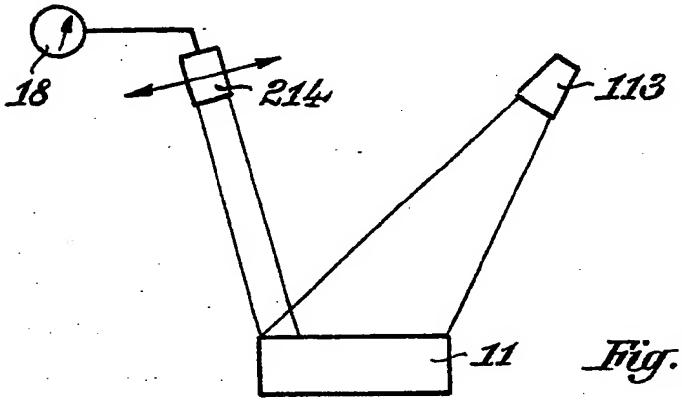

Fig. 6

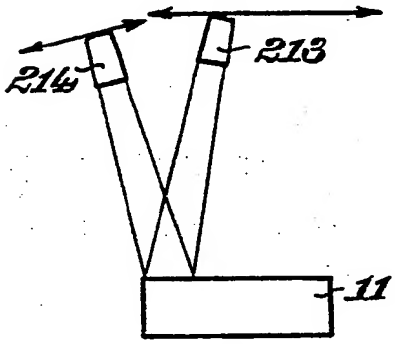

Fig. 7

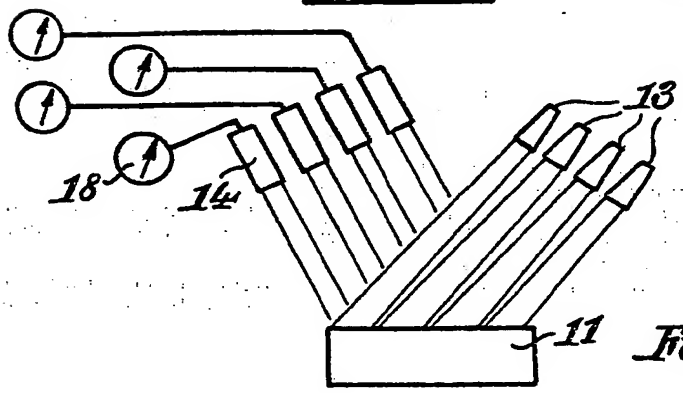

Fig. 8

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SHEETS 2 & 3

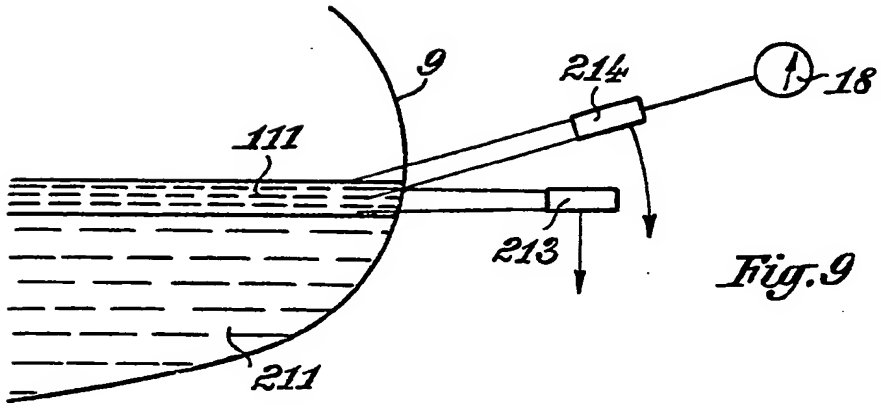

Fig.9

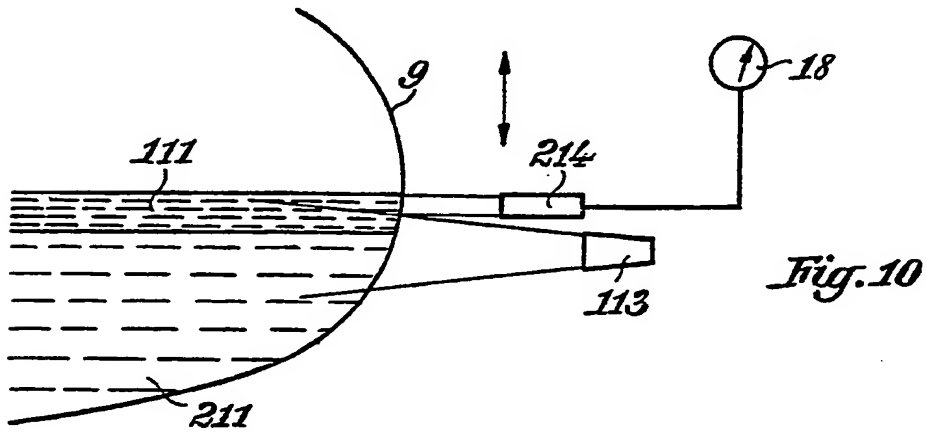

Fig.10

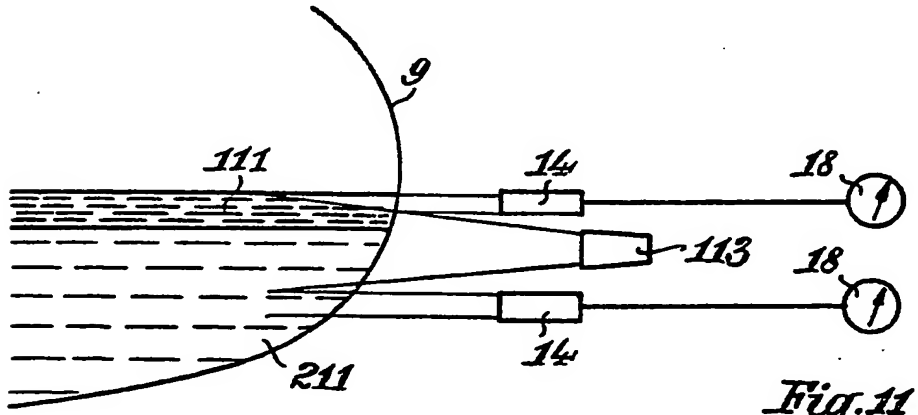

Fig.11

1,000,044 COMPLETE SPECIFICATION
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SHEETS 2 & 3

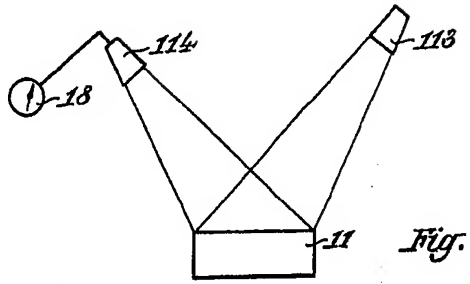

Fig. 5

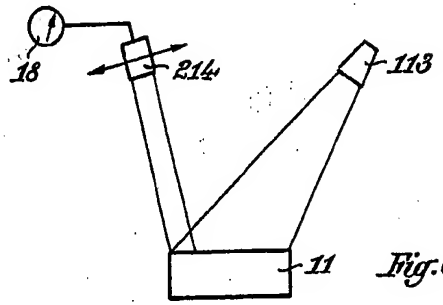

Fig. 6

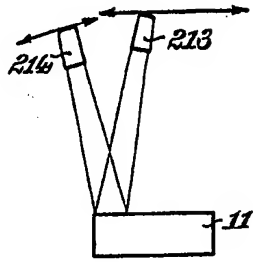

Fig. 7

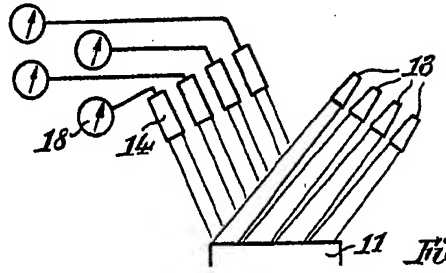

Fig. 8

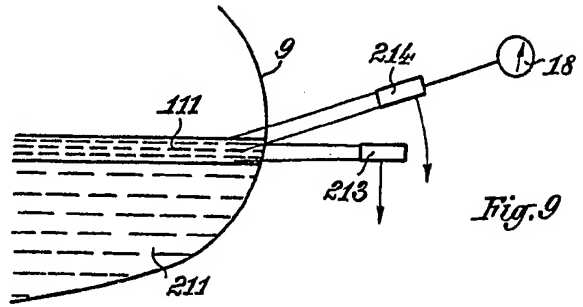

Fig. 9

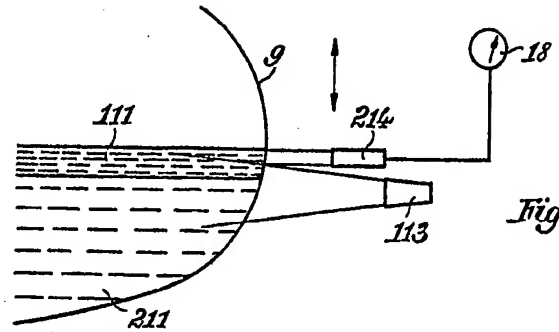

Fig. 10

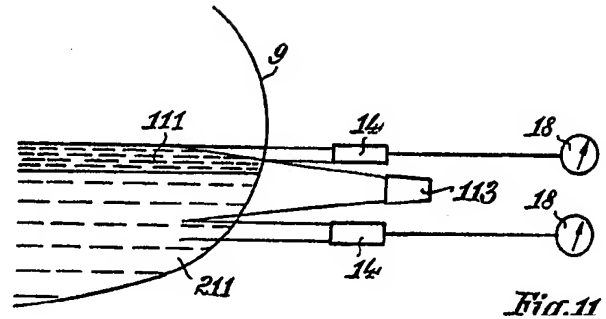

Fig. 11

PERGAMON

Applied Radiation and Isotopes 57 (2002) 573–577

Applied Radiation and Isotopes

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Coincidence measurement setup for PGAA and nuclear structure studies

P.P. Ember*, T. Belgya, J.L. Weil, G.L. Molnár

Institute of Isotope and Surface Chemistry, Hungarian Academy of Sciences, Chemical Research Centre, POB 77, H-1525 Budapest, Hungary

Received 28 March 2002; received in revised form 16 May 2002; accepted 28 May 2002

Abstract

A second cold-neutron beam experimental station has been built as part of the renewal of the PGAA facility of the Budapest Research Reactor. This new station has been instrumented for neutron-induced prompt γ-ray spectroscopy, involving γ–γ coincidence measurements. The experimental setup is discussed, and its performance and our data analysis method in a case of a radioactive source coincidence experiment are presented. © 2002 Elsevier Science Ltd. All rights reserved.

PACS: 25.40.Lw; 81.70.Jb; 82.80.Ej; 82.80.Jp

Keywords: (n, γ); E_n = cold; PGAA; Coincidence; Spectral interference

1. Introduction

The prompt gamma activation analysis technique (PGAA) (Molnár and Lindstrom, 1998; Molnár et al., 1997) is a rapidly developing analytical method. Many efforts have been made in various laboratories to improve its analytical sensitivity by reducing the background using well-collimated cold-neutron beams. A possible way of further improvement is to change the traditional Compton suppression detection technique for a more selective one. A natural choice is the use of the γ–γ coincidence technique with scintillator detectors (Gardner et al., 2000), or with HPGe detectors (Ember et al., 2002). The coincidence technique has been successfully applied for nuclear structure studies for several decades (Knoll, 2000, Chapter 12).

In a previous article (Ember et al., 2002) the regional γ–γ coincidence method for PGAA was introduced and compared with the traditional γ ray singles mode and the Compton suppressed mode of recording γ-ray spectra (Belgya et al., 1996; Molnár et al., 1997). That article contained only a brief description of the γ–γ coincidence experimental arrangement for neutron-induced prompt γ-ray spectroscopy (NIPS) studies. That arrangement complements our recently rebuilt PGAA facility at the 10 MW Budapest Research Reactor's cold-neutron laboratory. The complete facility will be described elsewhere (Revay et al., 2002). In our present article we give a detailed description of the γ–γ coincidence setup, its electronics, and the data handling methods. Its performance is presented in the results of a ^{60}Co decay γ–γ coincidence experiment.

2. The γ–γ coincidence arrangement

2.1. The NIPS setup

The γ–γ coincidence experimental setup is situated at the PGAA beam port of the Budapest Research Reactor, and it forms part of the NIPS facility. The experimental station is at the end of the No. 1 cold-neutron guide (Rosta et al., 2002) about 35.5 m distance

*Corresponding author. Fax: +36-1-392-2584.
E-mail address: ember@alpha0.iki.kfki.hu (P.P. Ember).

PII: S0969-8043(02)00137-9

from the reactor wall. As the neutrons leave the mirror guide, the full beam with $10 \times 2.5\,cm^2$ cross section is divided vertically by a two-hole ^6LiF loaded polymer collimator, located just before the main beam shutter. The two neutron beams then enter the PGAA (n,γ) experimental area through an Al tube system (Revay et al., 2002). The first beam divider is followed by several other similar collimators at key positions in the Al beam tube. The upper beam is used for the conventional Compton-suppressed PGAA analysis (Belgya et al., 1996; Molnár et al., 1997; Revay et al., 2002), while the lower beam passes through the PGAA target chamber and enters the NIPS target chamber. The latter is designed to satisfy diverse experimental conditions, including those of coincidence experiments.

The NIPS target chamber is positioned about 2.6 m downstream from the first collimator, and about 1 m from the PGAA target chamber. The housing is a square-shaped Al tube with an outer cross section of 5×5 cm and a wall thickness of 2 mm. This Al tube can be evacuated separately from the PGAA target chamber, and it encloses the neutrons as they travel from a collimator at its beginning to the beam stop. For neutron shielding, its inner walls are lined with a 3-mm thick layer of ^6LiF loaded polyethylene sheet. The collimated neutron beam has an approximately $2.5 \times 2.5\,cm^2$ cross section at the target position, with a measured thermal equivalent neutron flux of 3×10^7 n/cm^{2}s. The thin aluminum-windowed target chamber is lined with 2.3 mm thick sheets of highly enriched ^6LiF loaded polyethylene to protect the detectors from scattered neutrons. It can hold samples as large as 1.5 cm in diameter and 3.5 cm in length.

The small size of the target chamber allows us to use up to three closely positioned γ-ray detectors at one time, as shown in Fig. 1. All of them can be placed perpendicular to the neutron beam, one at each side, and one below the target chamber. The detector faces can be as close as 2.5 cm from the center of the target. Detectors can be either semiconductor or scintillation detectors. At present we can equip this experimental station with two coaxial HPGe detectors and a third planar HPGe detector, or with one HPGe and two fast BaF_2 detectors for fast timing experiments.

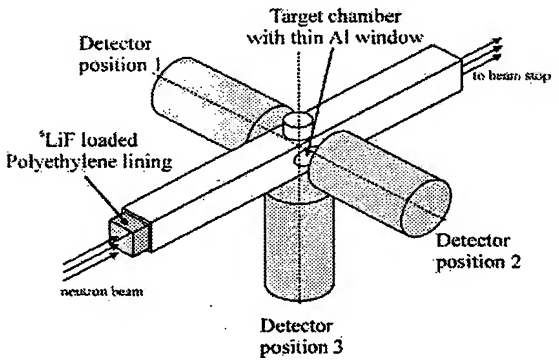

Fig. 1. The coincidence target chamber and the detector positions at the NIPS station.

For the demonstration of the performance of our γ–γ coincidence system we used two coaxial HPGe detectors. The larger detector (HPGe-I) had 30% efficiency relative to a $3'' \times 3''$ NaI detector and 1.8 keV resolution at 1332 keV γ-ray energy of ^{60}Co calibration source. The smaller detector (HPGe-II) had 15% efficiency and 1.9 keV resolution. The HPGe-II detector was placed horizontally at position 1, at a distance of 2.3 cm from the Al tube, and HPGe-I vertically at position 3, about 0.6 cm from the Al tube. Lead blocks of 5 cm thickness were used around the crystals as gamma shields, and plates of 1.5 mm thick lead were placed between the detectors and the tube to decrease the number of back-scattered γ-photons, and to filter out the X-rays.

2.2. The coincidence electronics

The electronics used in these γ–γ experiments are shown in Fig. 2. The energy signals of the detectors were shaped and amplified by spectroscopy amplifiers. The amplifier signals were digitized with 16k analog-to-digital converters (ADC). The time signals of both detectors were connected into fast filter amplifiers. The amplified and shaped time signals were plugged into two constant fraction discriminators (CFD), which were used in ARC timing mode. The CFD signal of the detector HPGe-I started the time-to-amplitude converter (TAC). The CFD signal of the detector HPGe-II was delayed by approximately 500 ns using a gate and delay generator, and it served as a STOP signal for the TAC. The TAC output of the time-to-amplitude converter was digitized in a homemade 4k ADC. The TAC single channel analyzer (SCA) logic output was connected into a level translator to be converted into one TTL and two NIM level signals. The TTL signal was used for gating the 4k ADC, which digitised the time spectrum. The NIM signals were fed into two segments of the gate and delay generator in order to synchronize them in time with the outputs of the spectroscopy amplifiers, and create the correct gating pulses for the two 16k ADCs that digitized the energy signals.

The energy signal of HPGe-II was also plugged into a fourth ADC without gating (not shown in Fig. 2). The data acquisition computer's MCA board collected this spectrum in singles mode. This spectrum was used as the neutron flux monitor of the system.

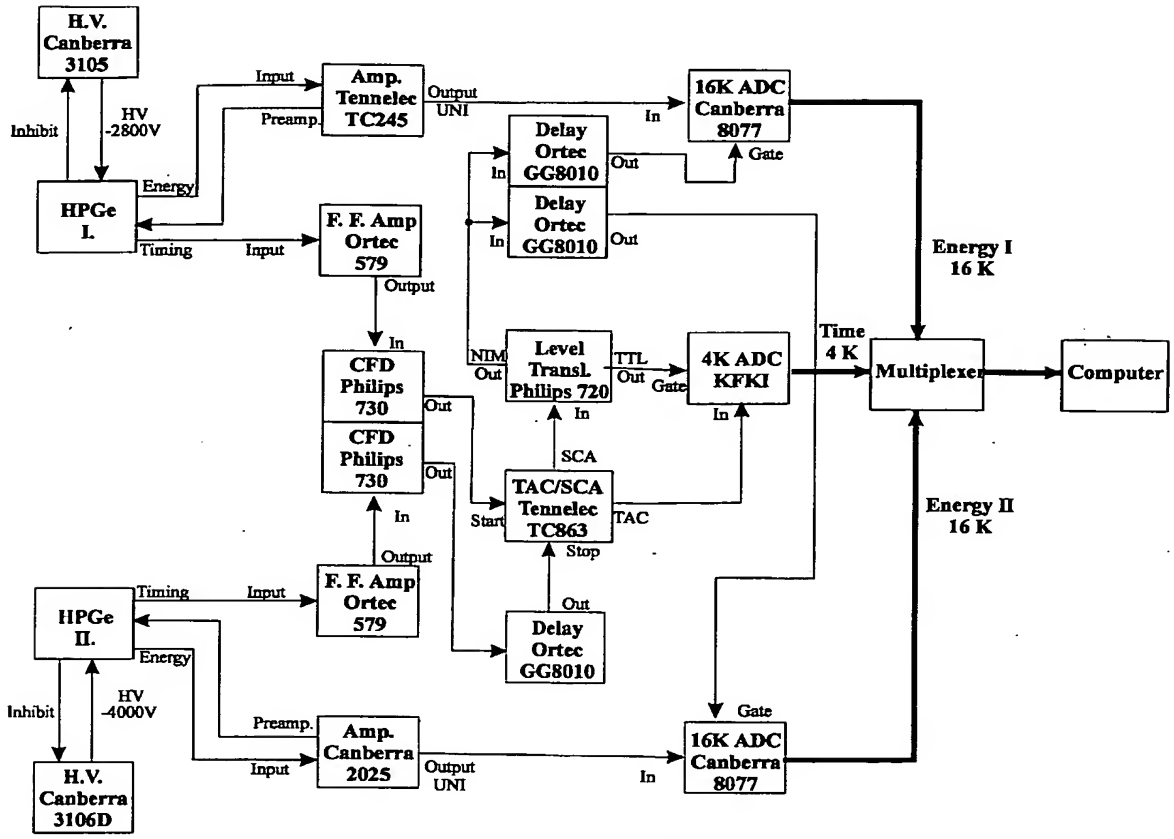

Fig. 2. The γ–γ coincidence electronics.

2.3. The multiplexer

The ADC data outputs were connected into a home-built multiplexer, which transferred the events to a PC-based data acquisition system. This data acquisition system was described briefly by Héjja et al. (Héjja et al., 1997); here we give some additional information about it.

The multiplexer has 10 input channels compatible with the data output of CANBERRA ADCs. Its two outputs are connected to a PC with two I/O cards: a National Instruments 32-bit AT–DIO card for the list mode data acquisition, and a 64 k-word Multi Channel Analyser (MCA) board, which is a product of KFA Jülich. The programmable multiplexer can be configured via its XILINX logic chips through the AT-DIO board. The multiplexer is able to serve both the list mode and MCA mode data collections at the same time. The multiplexer data-collecting time can be set in the range of 0–32768 ns for the list mode channels. This time is the waiting time for the other ADC outputs to arrive after the first signal arrived in any one channel. The collected data array is sent, after the waiting time has expired, to a PC via the AT-DIO card regardless of how many ADC outputs fired after the first one. Because of this, the list file can contain some data with no value. This is the so-called independent data collection mode. The dependent mode, where signals from all the ADCs have to be present, can be realized off-line with a simple program which removes all records with empty words from the list file. The independent mode is useful when double- and triple- or higher-fold events are to be collected at the same time.

3. Test measurement

The test measurement was performed off line with a ^{60}Co source of 7 kBq activity for 24 h. The de-exciting γ-rays have been measured in coincidence mode.

In this demonstration experiment about 2% of the records in the list mode data files contained one or two

blank words out of the three words making up a coincidence event. These are due to signals with amplitudes falling outside the range of one or more ADCs. After removing these unwanted events the resulting file contained 9 million coincidence events. The list file was processed off-line by homemade software, capable of creating projections of selected channels, while estimating the uncertainty and subtracting the background. We do not describe here the details of this program, but show its function through the example.

To illustrate the time resolution of our system, we present the time spectrum obtained in the ^{60}Co experiment. Gates were set on the 1173 keV γ-ray peak in the spectrum of HPGe-I and on the 1332 keV γ-peak in the spectrum of the HPGe-II detector. The resulting time spectrum is presented in Fig. 3. The time resolution is 12.7 ns full-width at half-maximum (FWHM) as shown in the figure. The peak-to-background ratio is about 500 to 1. These values are valid for a simple transition (with narrow energy gates) only.

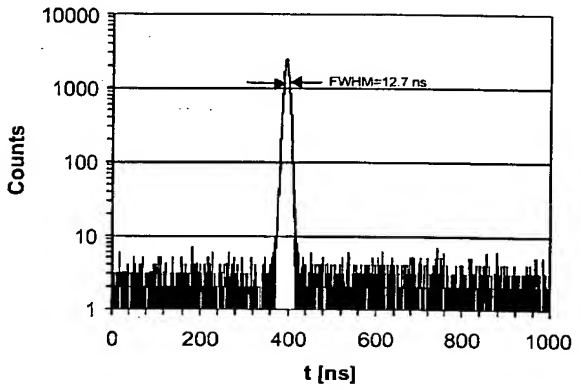

Fig. 3. Time spectrum of ^{60}Co γ–γ coincidence events. Gates were set on the 1173 and 1332 keV γ-peaks in the HPGe-I and HPGe-II energy spectra, respectively.

Fig. 4 presents the total projection and the gated cobalt spectra of the HPGe-I detector energy signals. For the latter, the 1332 keV γ-peak of the HPGe-II spectrum and the time peak in the time spectra were set as gates. To estimate the background, we set background regions on both sides of the time and energy peaks. The two background regions combined cover the same number of channels as the peak gates. The analysis program subtracts the projection of the backgrounds automatically from the coincidence projection spectra and also calculates the uncertainties in the gated spectrum. The calculated suppression of the 1332 keV γ-peak relative to the 1173 keV γ-ray peak was 485 in the presented case.

4. Summary

In this article we report about a new, second experimental station at our prompt gamma facility. The main purpose of this station is to perform (n,γ) experiments with a well-collimated cold-neutron beam. We present our γ–γ coincidence setup as one of its possible experimental uses, and have illustrated our coincidence data collection and analysis methods with the help of a test measurement.

In the future, we plan on performing more complex coincidence experiments on nuclei with less well-known level schemes. The background reduction capabilities of the coincidence setup is also expected to increase the

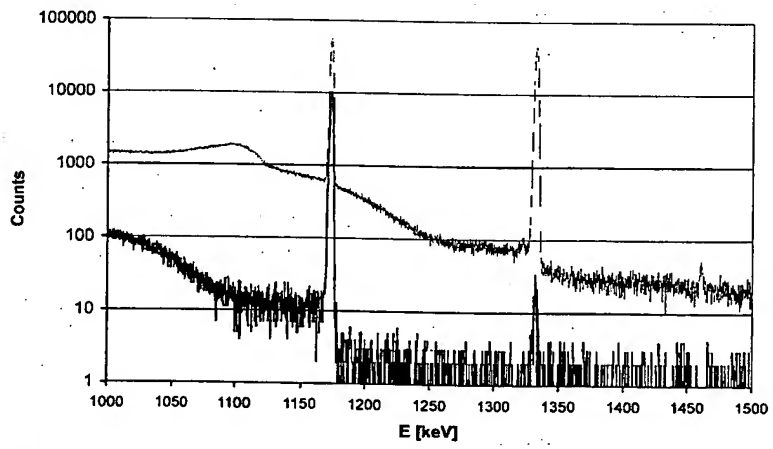

Fig. 4. HPGe-I detector energy spectra. Thin line: total projection, thick line: gated with the 1332 keV γ-peak of the HPGe-II spectrum and the time peak in the time spectra.

sensitivity for trace elements in Prompt Gamma Activation Analysis.

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